

#10

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE (Attorney Docket No. 97,845)

In re Application of:)	
F. Leo Hickey et al.)	
)	Examiner: R. Gorr
Serial No.: 09/289,043)	
)	Group Art Unit: 1711
Filed: April 9, 1999)	
)	
For: Pentane Compatible Polyester Polyols)	
)	

DECLARATION OF F. LEO HICKEY

Commissioner for Patents and Trademarks Washington, D.C. 20231

Sir:

- I, F. Leo Hickey, of 215 S. Chestnut Avenue, Arlington Heights, IL 60005, declare as follows:
- 1. I received a B. Sc. degree in Chemistry from Lehigh University, Bethlehem, PA, in 1968.
- 2. I presently am employed as a Senior Research Chemist at Stepan Co., Northfield, IL.
- 3. From 1964 until the present time, I have been employed in various research positions at Stepan Co., Northfield, IL.
- 4. I am the author or co-author of two papers, one related to agricultural chemicals, and the other related to polyurethanes.

- 5. Because of my extensive background in emulsifiers, surfactants, and polyols, I consider myself an expert in the area of polyols and their use in forming polyurethane and polyisocyanurate foams. I am inventor of this application and submit this declaration in support of the patentability of my invention.
- 6. I have reviewed the Office Action mailed October 19, 2000, and the pending claims in the above-identified application and understand that the Examiner has rejected the claims under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No. 4,644,048, Magnus et al. ("Magnus") in view of van der Wouden, "The Use of Polyester Polyols in CFC-Free Rigid Foams," Utech '94, Paper 21, pp. 1-5 ("van der Wouden (Utech 94)"). I submit that Magnus in view of van der Wouden (Utech 94) does not suggest to an ordinary skilled artisan a reasonable expectation of success in making the compositions of the instant claims.
- 7. Van der Wouden (Utech 94) teaches that low polarity polyols are needed for hydrocarbon compatibility. In the Office Action, the Examiner concludes that the hydrophobic component of

Magnus would lower the polarity sufficiently to enable compatibility with the hydrocarbon blowing agent.

- 8. In van der Wouden (Utech 94), three types of dimer acid-based polyester polyols were added to a hydrocarbon-incompatible polyether polyol. At constant polyether/polyester ratio, hydrocarbon compatibility increased as polyester polyol polarity decreased.
- 9. Van der Wouden (Utech 94) identifies the polyester polyol enabling best hydrocarbon solubility as dimer acid-based "Unichema polyester OHV 345." See page 2, column 1, line 52. Unichema sells this polyester polyol, which has a hydroxyl value ("OHV") of 345, under the tradename Priplast 3185. See van der Wouden (Utech Asia 95), page 2, column 1, line 54. Van der Wouden, "Performance of Oleochemical Based Polyester Polyols in Polyurethanes," Utech Asia '95, Paper 34, pp. 1-7 ("van der Wouden (Utech Asia 95)") at pages 2-3 identifies Priplast 3181 as a polyester polyol having hydrocarbon compatibility even better than that of Priplast 3185.
- 10. Although neither van der Wouden (Utech 94) nor Unichema identifies how much of the hydrophobic dimer acid is present in

the polyester polyols which provide hydrocarbon compatibility, one of ordinary skill in the art can estimate the dimer acid content of a given polyester polyol using various methods.

- 11. Van der Wouden (Utech 94) characterizes the Unichema polyester polyols studied as being based on a dimer acid esterified "with polyalcohols." See van der Wouden (Utech 94), page 2, column 1, line 1. Van der Wouden (Utech Asia 95) characterizes the investigated Unichema polyester polyols as being derived from transesterification of dimer acid "with polyols such as glycerine." See van der Wouden (Utech Asia 95), page 1, column 2, line 25. Therefore, for the purpose of estimating dimer acid content, it would be reasonable for one familiar with polyester polyols to assume Priplast 3181 and Priplast 3185 are reaction products of dimer acid and glycerol.
- 12. One method of estimating dimer acid content is by examining the relative densities of the reactants and the product. The density of glycerol is 1.261 g/ml. See Aldrich Chem. Co. catalog, 1996-97, page 759. The Unichema publications list the density of dimer acid (Pripol 1008), polyester polyol Priplast 3185, and polyester polyol Priplast 3181 as 0.95 g/ml, 1.05 g/ml, 1.07 g/ml, respectively. See

High Purity Dimer Fatty Acids In Condensation Polymers, Unichema Int'l, October 1989; Priplast Polyester Polyols for the Polyurethane Industry, Unichema Int'l, no date available; Priplast Polyester Polyols for the Polyurethane Industry, Unichema Int'l, May 1995. Because there is a considerable difference between the densities of the two reactants (dimer acid and glycerol), and the only loss on reaction is water (a relatively small molecule), one of ordinary skill would reasonably assume that the density of the reaction product can be approximated by the sum of the weight-averaged densities of the reactants, as follows:

 $\chi_{i} = \chi_{i} = \infty$

reaction product density = (weight % glycerol x density of glycerol) + (weight % dimer acid x density of dimer acid).

Conversely, once the density of the reaction product is known, the approximate weight percentages of the reactants can be calculated. For the Priplast 3185 of van der Wouden (Utech 94), the above calculation produces the following estimate of dimer acid content:

(1.261-1.05)/(1.261-0.95) = 0.68, or <u>68% dimer acid</u>.

For the Priplast 3181 of van der Wouden (Utech Asia 95), the corresponding calculation is:

(1.261-1.07)/(1.261-0.95) = 0.61, or <u>61% dimer acid</u>.

13. An alternative method for estimating dimer acid content involves examining both the stoichiometry of the reaction that produces the polyol and the properties of the reaction product polyol. If one mole of dimer acid (Pripol 1008, average acid value 196, equivalent weight 286, molecular weight 572) is fully esterified with 2 moles of glycerol (molecular weight 92) to generate a polyol, the dimer acid comprises 76 percent by weight based on the total weight of reactants. However, the expected hydroxyl value of such a reaction product is only 312, compared to quoted average hydroxyl values of 345 for Priplast 3185 and 445 for Priplast 3181. If enough excess glycerol (hydroxyl value 1827) is used in the reaction to bring the final hydroxyl values to 345 and 445, respectively, the compositions are:

For Priplast 3181:

8.8% by wt. free glycerol

91.2% by wt. dimer acid/glycerol ester

For Priplast 3185:

2.2% by wt. free glycerol

97.8% by wt. dimer acid/glycerol ester

These final compositions would theoretically require the following amounts of starting materials:

For Priplast 3181:

572 parts by wt. dimer acid

184 parts by wt. glycerol to enter into the esterification

70 parts by wt. free glycerol

For Priplast 3185:

572 parts by wt. dimer acid

184 parts by wt. glycerol to enter into the esterification

16 parts by wt. free glycerol

The percentages of dimer acids in these mixtures are:

Priplast 3181: 69% dimer acid

Priplast 3185: 74% dimer acid

14. Both estimation methods suggest that the content of hydrophobic component required for enabling hydrocarbon

compatibility is well over 50 percent by weight. Thus, after considering van der Wouden (Utech 94), an ordinary skilled artisan would expect that the amount of hydrophobic soybean oil required to produce hydrocarbon compatibility in an otherwise polar phthalic anhydride-based polyol, like those of Magnus, is also likely to be well over 50 percent by weight.

- 15. The need described by van der Wouden (Utech 94) to use very high hydrophobic content to compatibilize the hydrocarbon blowing agent would lead one to expect the use of low levels of hydrophobic material to lead to a lack of hydrocarbon compatibility; i.e., low levels, i.e., from about 1 to about 40% by weight, of hydrophobic material would be expected to be unacceptable for compatibilizing the hydrocarbon blowing agent. Instead, however, the level of hydrophobic material of about 1 to about 40% required in the claims leads to polyols that are unexpectedly found to be acceptable in making foams with hydrocarbon blowing agents.
- 16. In view of the fact that the above analysis, which is based on information available at the time of the invention, indicates that the van der Wouden (Utech 94)

approach requires excessively high levels of reacted hydrophobic materials, it is surprising to find that much lower levels of these materials are effective in compatibilizing high percentages of hydrocarbons. The ability of the claimed compositions to use lower levels of monofunctional hydrophobic residues avoids the problem of excessively reducing functionality and the expected possibility of resulting inferior polymer properties.

17. Van der Wouden (Utech 94) teaches that well over 50 percent by weight hydrophobic component is required for enabling hydrocarbon compatibility. Magnus is silent on the issue of hydrocarbon compatibility, and teaches only that incorporating up to 28 mole percent of hydrophobic component into a polyester polyol enables fluorocarbon compatibility. U.S. patent No. 4,644,048, Magnus et al., col. 6, line 31. The most preferred range of hydrophobic compound in Magnus is 5 to 15 mole percent. Magnus, col. 7, line 37. Magnus examples show hydrophobic component being utilized at levels of about 20 percent by weight. Magnus, Table X, col. 22, Example 16 (decyl alcohol used at 19.1 wt%); col. 21, Example 9 (soybean oil used at 20.6 wt%). The claimed invention enables hydrocarbon compatibility even though it incorporates

no more than about 40 percent by weight hydrophobic component. An ordinary skilled artisan considering Magnus in view of van der Wouden (Utech 94) would not expect that incorporating from about 1 to about 40 percent by weight or less of hydrophobic component would sufficiently lower the polarity of the polyester polyol to enable compatibility with a hydrocarbon blowing agent.

18. I declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the U.S. Code and that such willful false statements may jeopardize the validity of this application and any patent issuing thereon.

Respectfully submitted,

Dated: 3/19/01

F. Leo Hickey